PART II

Volatile/Semivolatile Data Validation Fuctional Guidelines

December 1996

VOLATILE/SEMIVOLATILE DATA VALIDATION FUNCTIONAL GUIDELINES - PART II

The requirements to be checked in validation are listed below. "CCS" indicates that the contractual requirements for these items will also be checked by Contract Compliance Screening (CCS). CCS requirements are not always the same as data validation criteria. "CADRE" indicates that CADRE checks for these items in CLP-Low/Medium Organic electronic data and provides a CADRE printout. Additional manual evaluation may be required. Refer to the Guidance Document for Completing Region I Data Validation Utilizing CADRE Data Review, February 1995, or most recent revision (Attachment L of Part I, Data Validation Manual).

Appendices

I. PRESERVATION AND TECHNICAL HOLDING TIMES

A. OBJECTIVE

The objective is to ascertain the validity of the analytical results based on the preservation techniques which were used and the holding time of the sample from time of collection to time of sample preparation and sample analysis, as appropriate.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. REGION I PRESERVATION CRITERIA

SAMPLE TYPE	PRESERVATION CODE
Volatile Aqueous [a]	1,2,3
Volatile Soil/Sediment [b]	1,3
Semivolatile Aqueous [a]	1,3
Semivolatile Soil/Sediment [b]	1,3
VOA/SV Sludge [b]	1,3
VOA/SV Oily Waste [b]	1,3
VOA/SV Biological Tissue [c]	3,4
VOA Air (Canister) [c]	3,5
VOA Air (Adsorbent Tubes) [c]	1,3
SV Air (PUF, Filters) [c]	1,3
SV Wipes [c]	1,3
SV Fly Ash [b]	1,3

Preservation Code:

1. Cool @ 4EC (± 2E)
2. Preserve with HCl to at least pH 2
3. Protect from light
4. Freeze
5. Room Temperature (Avoid excessive heat)

References:

a. 40 CFR, Part 136, Appendix A, 600 Series

b. SW-846, 8000 Series

c. Region I policy

2. REGION I TECHNICAL HOLDING TIME CRITERIA

SAMPLE TYPE	CRITERIA
Volatile Aqueous [a]	If the sample was not properly preserved with HCl but was protected from light and stored at 4EC (± 2E), aromatic volatiles must be analyzed within 7 days and non-aromatic volatiles within 14 days of sample collection. If the sample was properly preserved, then both aromatic and non-aromatic volatiles must be analyzed within 14 days of sample collection.
Volatile Soil/Sediment [b]	Properly preserved soil/sediment samples must be analyzed within 14 days of sample collection.
Semivolatile [a] Aqueous	Extraction of properly preserved aqueous samples by liquid-liquid procedures must be started within 7 days of sample collection. Extraction of properly preserved aqueous samples by separatory funnel or solid phase extraction (SPE) must be completed within 7 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.
Semivolatile Soil/Sediment [b]	Extraction of properly preserved soil/sediment samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.
VOA/SV Sludge [b]	Purge and trap or extraction of properly preserved sludge samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.

VOA/SV Oily Waste [b]	Purge and trap or extraction of properly preserved oily waste samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.

SAMPLE TYPE	CRITERIA
VOA/SV Biological Tissue [c]	Extraction and analysis of frozen tissue must be completed within 60 days of sample collection. Tissue must remain frozen until homogenization is completed. Extraction and/or analysis must be initiated immediately after homogenization.
VOA Air [c]	Analyses of properly preserved VOA air samples must be completed within 14 days of sample collection. Pre-cleaned and certified volatile air collection devices, i.e., Tenax and charcoal cartridges and SUMMA canisters, must be utilized for sample collection within the method-specified time frame.
SV Air [c]	Analyses of properly preserved SV air samples must be completed within 14 days of sample collection. Pre-cleaned and certified semivolatile air collection devices, i.e., PUFS, and filters, must be utilized for sample collection within the method-specified time frame.

SV Wipes [c]	Extraction of properly preserved SV Wipe samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.
SV Fly Ash [b]	Extraction of properly preserved SV fly ash samples by sonication or soxhlet procedures must be completed within 14 days of sample collection. Extracts must be analyzed within 40 days following sample extraction.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
1. Volatile Samples a. Preservation Examine the sample records (EPA Traffic Reports and/or COC Forms), Sample Receipt forms (DC-1 Form), laboratory tracking/storage forms, and the data package narrative to verify that samples were properly preserved by the sampler and the laboratory maintained preservation. If adequate documentation on field sample preservation is not present in the data package, then the validator must contact the sampler and/or laboratory to obtain the missing information. i. Verify that volatile samples were refrigerated or frozen (as required) and protected from light according to Region I preservation criteria.	All potential impacts on the sample data resulting from preservation and/or holding time anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum. 1. Volatile Samples a. Preservation If the sampler cannot be contacted or cannot produce adequate preservation documentation, then the validator should assume the samples were not preserved and should document on the holding time worksheet the date that sampler contact was attempted and/or established. If the laboratory cannot provide adequate sample preservation information, then the validator should use professional judgment to accept, qualify or reject the sample data. If the samples were not preserved properly in the field and/or if the laboratory failed to properly maintain sample preservation, then the validator should take the following actions: i. If volatile samples for aqueous and soil/sediment matrices were not refrigerated and/or protected from light according to Region I preservation criteria, then the validator should estimate (J) positive detects and reject (R) non-detects for the affected samples, regardless of whether or not technical holding time criteria were met and regardless of whether or not the sample (aqueous) was acid preserved. For other matrices, the validator should estimate (J) positive detects and should use professional judgment to qualify or reject non-detects when temperature and light protection preservation criteria were not met. Professional judgment should be used when the

C. EVALUATION	D. ACTION
1. a. ii. Verify from the EPA Traffic Report and/or COC Form and the data package narrative that aqueous volatile samples were preserved with HCl according to Region I preservation criteria.	1. a. ii. If data package documentation does not list the pH of each aqueous VOA sample, then the validator should contact the laboratory to obtain any omitted information. If aqueous volatile samples were not preserved with HCl according to Region I preservation criteria, then the validator must evaluate holding times to determine if qualification of sample data is necessary for detected and non-detected aromatic and non-aromatic compounds.
iii. Review sample records (COC Forms, Sample Receipt and/or Login Forms, DC-1, etc.) to determine if excessive headspace in any aqueous sample was noted by the laboratory.	iii. If volatile aqueous samples contain excessive headspace (bubbles greater than 2 mm diameter should not be present), then the validator should estimate (J) positive detects and reject (R) non-detects.
b. Technical Holding Times	b. Technical Holding Times
i. Verify that volatile samples were analyzed within the technical holding time criteria. Establish technical holding times by comparing sampling dates reported on the EPA Traffic Report and/or COC Forms with dates of analysis on tabulated result forms.	i. If aqueous volatile samples were not preserved with HCl (but refrigeration and light protection criteria were met) and the samples were not analyzed within 7 days, then the validator should: - Estimate (J) aromatic positive detects analyzed within 14 days. - Reject (R) aromatic non-detects. - Accept non-aromatic positive detects analyzed within 14 days - Accept non-aromatic non-detects analyzed within 14 days. - Estimate (J) aromatic positive detects analyzed after 14 days. - Estimate (J) non-aromatic positive detects analyzed after 14 days. - Estimate (UJ) non-aromatic non-detects analyzed after 14 days. If volatile samples for aqueous and soil/sediment

C. EVALUATION	D. ACTION
*1. b. ii. Check the raw data including instrument run and extraction logs to verify reported sample extraction and analysis dates.	1. b. ii. If discrepancies between the raw data and reported data are found, then the validator should contact the laboratory to obtain corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
2. Semivolatile Samples a. Preservation Examine the sample records (EPA Traffic Reports and/or COC Forms), Sample Receipt forms (DC-1 Form), laboratory tracking/storage forms, and the data package narrative to verify that samples were properly preserved by the sampler and the laboratory maintained preservation. If adequate documentation on field sample preservation is not present in the data package, then the validator must contact the sampler and/or laboratory to obtain the missing information.	2. Semivolatile Samples a. Preservation If the sampler cannot be contacted or cannot produce adequate preservation documentation, then the validator should assume the samples were not preserved and should document on the holding time worksheet the date that sampler contact was attempted and/or established. If the laboratory cannot provide adequate sample preservation information, then the validator should use professional judgment to accept, qualify or reject the sample data. If the samples were not preserved properly in the field and/or if the laboratory failed to properly maintain sample preservation, then the validator should take the following actions:
i. Verify that semivolatile samples were refrigerated or frozen (as required) and protected from light according to Region I preservation criteria.	i. If semivolatile samples for aqueous and soil/sediment matrices were not refrigerated and/or protected from light according to Region I preservation criteria, then the validator should estimate (J) positive detects and estimate (UJ) non-detects for the affected samples, regardless of whether or not technical holding time criteria were met. For other matrices, the validator should estimate (J) positive detects and

C. EVALUATION	D. ACTION
2. b. Technical Holding Times i. Verify that semivolatile samples were extracted within technical holding time criteria. Establish extraction holding times by comparing sampling dates reported on the EPA Traffic Report and/or COC Forms with dates of extraction reported on tabulated result forms. 1. Verify that liquid-liquid extractions for semivolatile aqueous samples were begun within 7 days of sample collection. 2. Verify that aqueous semivolatile extractions by separatory funnel were completed within 7 days of sample collection. (Note: OLM03.2 does not allow separatory funnel extraction of semivolatiles.) 3. Verify that aqueous semivolatile extractions by solid phase extraction (SPE) or other extraction technique were completed within 7 days of sample collection. 4. Verify that semivolatile soil/sediment sample extractions by sonication or soxhlet procedures were completed within 14 days of sample collection. 5. Verify that samples of other matrices, i.e., wipes, biological tissue, were extracted within the Region I holding time criteria. Verify that semivolatile samples and/or extracts (as	2. b. Technical Holding Times i. If aqueous and soil/sediment semivolatile samples were properly preserved, but the technical extraction and/or analytical holding time criteria were exceeded, then the validator should estimate (J) positive detects and estimate (UJ) non-detects. For other matrices, the validator should estimate (J) positive detects and should use professional judgment to qualify or reject non-detects when technical holding time criteria are exceeded. For all matrices, if semivolatile extraction technical holding time criteria were grossly exceeded (> 28 days) and/or analytical technical holding time criteria were grossly exceeded (> 60 days), then the validator should estimate (J) positive detects and reject (R) non-detects.

C. EVALUATION			D. ACTION	
*2.	b. ii.	Check the raw data including extraction and instrument run logs to verify reported sample extraction and analysis dates.	2.	b. ii. If discrepancies between the raw data and reported data are found, then the validator should contact the laboratory to obtain corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.1.b.ii, C.2.b.ii

Table VOA/SV-I-1:

QUALIFICATION OF VOLATILE ANALYTES BASED ON PRESERVATION & TECHNICAL HOLDING TIMES

PRESERVATION			TECHNICAL HOLDING TIMES			
Matrix	Refrig. & Light Protected	Acid Preserved	# 7 Days	7 < HT # 14 Days	14 < HT # 28 Days	> 28 Days
AQ	No	Yes or No	J - detects R - non-detects	J - detects R - non-detects	J - detects R - non-detects	J - detects R - non-detects
AQ	Yes	Yes	A	A	J - detects UJ - non-detects	J - detects R - non-detects
AQ	Yes	No	A	**Aromatics** J - detects R - non-detects **Non-aromatics** A - detects A - non-detects	**Aromatics** J - detects R - non-detects **Non-aromatics** J - detects UJ - non-detects	J - detects R - non-detects
S/S	No	N/A	J- detects R - non-detects	J - detects R - non-detects	J - detects R - non-detects	J - detects R - non-detects
S/S	Yes	N/A	A	A	J - detects UJ - non-detects	J - detects R - non-detects

Note: AQ = Aqueous, S/S = Soil/Sediment

For other matrices, the validator should estimate (J) positive detects and use professional judgment to qualify or reject non-detects when Region I preservation and/or technical holding time criteria are not met.

For VOA aqueous samples containing excessive headspace (bubbles greater than 2 mm diameter); J-detects, R-non-detects

Table VOA/SV-I-2:

QUALIFICATION OF SEMIVOLATILE ANALYTES BASED ON PRESERVATION & TECHNICAL HOLDING TIMES

PRESERVATION		TECHNICAL HOLDING TIMES		
Matrix	Refrig. & Light Protected	Extracted and/or Analyzed Within H.T.	Extracted and/or Analyzed Outside H.T.	If Extraction HT > 28 days and/or Analytical HT > 60 days
AQ and S/S	Yes	A	J - detects UJ - non-detects	J - detects R - non-detects
AQ and S/S	No	J - detects UJ - non-detects	J - detects UJ - non-detects	J - detects R - non-detects

Note: AQ = Aqueous, S/S = Soil/Sediment

For other matrices, the validator should estimate (J) positive detects and use professional judgment to qualify or reject non-detects when Region I preservation and/or technical holding time criteria are not met.

REFERENCES

a - 40 CFR, Part 136, Appendix A, 600 Series

b - SW-846, 8000 Series

E. EXAMPLES

Example #1: (Improper preservation (without acid); Analysis holding time exceeded)

Aqueous volatile sample SAA99 was analyzed by routine analysis following CLP SOW OLM03.2. The validator examines the data package narrative and determines that the laboratory did not report the pH. The validator contacts the laboratory to determine whether the pH was checked by the laboratory and notes that it was not checked. The validator then examines the Traffic Report contained in the data package and notes that the sampler failed to record what, if any, preservation techniques were utilized. The validator attempts, but fails, to contact the sampler. It cannot be determined if the sample was preserved by the sampler with HCl.

The sampling date for SAA99 was 6/1/95 and the analysis date was 6/21/95, 20 days from sampling. The aqueous volatile samples exceeded the technical holding time criteria for aromatics and non-aromatics. The validator examines the Form I and notes that benzene, toluene, ethylbenzene, chlorobenzene, and xylenes (aromatics) are not detected and that acetone (non-aromatic) is reported at 30 ug/L. The validator reports the benzene, toluene, ethylbenzene, chlorobenzene, and xylenes non-detects as rejected (R), the non-aromatic non-detects as (UJ), and acetone as 30J on the Data Summary Table. The validator notes in the Data Validation Memorandum that the sample data are qualified based on improper preservation (without acid) and exceeded technical holding times.

Example #2: (Improper preservation (refrigeration); Holding times met)

Volatile air samples SAA11-SAA22 were analyzed by the most recent Region I analytical specification for Method TO-1. The laboratory noted in the data package narrative that the samples were received on a Friday afternoon and remained unrefrigerated in the shipping area for over 2 days. The laboratory further noted that this area

has no climate control and that temperatures routinely exceed that of the sample storage area by 15-20EC. The validator uses professional judgment to estimate (J) positive detects and reject (R) non-detects in all samples on the Data Summary Table due to the exposure to excessive heat over the 2 day period and discusses this problem in the Data Validation Memorandum.

Example #3: (Proper preservation; Analysis holding time exceeded)

Volatile soil sample SAA33 was sampled on 8/1/95 and was received at the laboratory on 8/2/95. Upon examination of the Traffic Report and the laboratory sample receipt and tracking information, the validator determines that the sample was shipped and stored at 4EC and was light protected. As noted in the data package narrative, due to a laboratory tracking error, the laboratory analyzed the sample following CLP SOW OLM03.2 on 8/18/95, 17 days from the sampling date. The validator estimates (J) the positive detects of sample SAA33 and estimates (UJ) the non-detects on the Data Summary Table and discusses this problem in the Data Validation Memorandum.

Example #4: (Proper preservation; Extraction holding time grossly exceeded)

Semivolatile soil sample SAA44 was sampled on 8/1/95 and received at the laboratory on 8/2/95. Upon examination of the Traffic Report, laboratory receipt information, and sample tracking records, the validator determines that the sample was properly preserved at 4EC and was light protected. The sample was not extracted until 9/1/95, 31 days from sampling date, due to a laboratory tracking error and extraction holding times were grossly exceeded. The validator estimates (J) the positive detects of sample SAA44 and rejects (R) the non-detects on the Data Summary Table and discusses this problem in the Data Validation Memorandum.

II. GC/MS INSTRUMENT PERFORMANCE CHECK (TUNING)

A. OBJECTIVE

Gas chromatograph/mass spectrometer (GC/MS) instrument performance (tuning) checks are performed to ensure proper mass calibration and resolution, identification and to some degree, sensitivity.

B. CRITERIA

GC/MS instrument performance (tuning) criteria are not sample specific. Since conformance is determined using standard materials, these criteria should be met under all circumstances. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
1. Verify from the reported results that the mass scale is correct (amu assignments are accurate) and that the ion abundance QC acceptance criteria specified in the method were met for each 12-hour period that samples were analyzed.	All potential impacts on the sample data resulting from tuning anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum. 1. a. If tabulated result forms are not present for each 12-hour period for which samples are analyzed, then the validator should contact the laboratory to obtain the tabulated forms. b. If the mass scale is incorrect and amu assignments are inaccurate, then the validator should reject (R) all data associated with that tune. The data should be returned to the laboratory and payment denied. c. If ion abundance QC acceptance criteria are not met, then professional judgment should be used to determine to what extent the data may be utilized. The most important factors to consider are the empirical results that are unrelated to retention time and type of instrumentation.

C. EVALUATION	D. ACTION
*2. Compare the reported tuning results on each GC/MS Tuning and Mass Calibration Form with each raw data mass listing and mass spectrum submitted. Verify that the laboratory has not made any transcription or calculation errors.	2. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*3. If possible, verify that spectra were generated using appropriate background subtraction techniques. Since the spectra are obtained from chromatographic peaks that should be free from coelution problems, background subtraction actions resulting in spectral distortions for the sole purpose of meeting the contract or method specifications are contrary to quality assurance objectives and are, therefore, unacceptable.	3. If the validator has reason to believe that tuning/instrument performance checks were achieved using non-compliant techniques, then the performance and procedures of the laboratory merit further investigation.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.2, C.3

E. EXAMPLES

Example #1: (Ion abundance criteria not met for several ions)

The validator examines tabulated and raw tuning data generated under CLP SOW OLM03.2 to check for calculation and transcription errors. The validator compares the BFB mass spectrum and mass listing with Form V-A. The ion abundances have not been normalized to ion 95 as per the SOW and, when normalized by the validator, do not meet the SOW ion abundance criteria. The validator notes that the abundance criteria for ions 50, 75, 96, and 174 are exceeded by 25%. The validator uses professional judgment to estimate (J) all positive detects and estimate (UJ) all non-detects on the Data Summary Table for samples associated with that tune and discusses this problem in the Data Validation Memorandum.

Example #2: (Ion abundance criteria not met for one ion)

The validator examines tabulated and raw tuning data generated under CLP SOW OLM03.2 to check for calculation and transcription errors. The validator compares the DFTPP mass spectrum and mass listing with Form V-B. The % Relative Abundance for ion 275 is 35% of ion 198 (OLM03.2 criteria for ion 198 is 10.0 - 30.0% of mass 198). The validator uses professional judgment to accept the tune since only one ion abundance slightly exceeds criteria. The validator reviews the mass spectra for all positive hits in samples in accordance with Section XII, Target Compound Identification and determines that all ion abundance ratios are acceptable. The validator discusses the non-compliant tune and justifies the decision to accept the sample data in the Data Validation Memorandum.

Example #3: (Mass calibration error)

The validator examines tabulated and raw DFTPP tuning data generated following method 625 to check for calculation and transcription errors. The validator notes that the tabulated tuning results were acceptable, however, the raw data do not agree with the tabulated results. Upon further review of the raw data, the validator notes that the mass calibration is off by 1 amu. In addition, surrogate recoveries and internal standard areas were unacceptably low. The validator rejects (R) all associated data, returns the data package to the laboratory, and payment is denied. The EPA Site Manager is informed by letter and resampling is subsequently scheduled.

III. INITIAL CALIBRATION

A. OBJECTIVE

Compliance requirements for initial calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Initial calibration data demonstrate that the instrument is capable of satisfactory performance at the beginning of the analytical sequence by producing a linear calibration curve.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. Initial calibration standards containing volatile and semivolatile target and surrogate compounds at method-specific concentrations are analyzed prior to the analysis of any field samples, QC samples, and blanks, or as necessary if the continuing calibration method acceptance criteria are not met. The initial calibration and any associated field samples, QC samples, and blanks must be analyzed within 12 hours of the associated GC/MS instrument performance check.

2. Initial calibration standards must be analyzed using the same instrumental conditions that will be used to analyze field samples, QC samples, and blanks.

3. The mean Relative Response Factors (RRFs) for all volatile and semivolatile target and surrogate compounds in each initial calibration must be greater than or equal to 0.05.

   The Percent Relative Standard Deviation (%RSD) for all volatile and semivolatile target and surrogate compound RRFs in each initial calibration must be less than or equal to 30.0 percent.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from initial calibration anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. a. Verify that the initial calibration standards were analyzed at the method-required concentrations and frequency, and that the standards were analyzed within 12 hours of the associated GC/MS instrument performance check.	1. a. If the laboratory did not use the required concentrations and/or frequency when analyzing the initial calibration standards, or the standards were not analyzed within 12 hours of the associated GC/MS instrument performance check, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected.
b. Verify that the method-required calibration standard(s) was used for calculating sample results if any sample results were calculated using an initial calibration.	b. If the correct method-required calibration standard(s) was not used to quantitate sample results, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*2. Verify that the same instrument parameters were used for sample and calibration analyses, and that the instrument parameters which were utilized met method requirements.	2. If correct instrument parameters (i.e., purge and trap conditions, etc.) were not used for the initial calibration standards and sample analyses, then the validator should contact the laboratory to obtain corrected data and forms. a. If the laboratory is unable to submit a correct initial calibration, then the validator should determine whether a qualitative analysis is of any benefit by reviewing the project Data Quality Objectives. b. If the data are deemed unusable, then the validator should reject (R) all associated data. The data should be returned to the laboratory and payment denied.

C. EVALUATION	D. ACTION
3. Verify that the RRFs for all volatile and semivolatile target and surrogate compounds are greater than or equal to 0.05 in the initial calibration. Verify that the %RSDs for all volatile and semivolatile target and surrogate compound RRFs do not exceed 30.0% in the initial calibration. Evaluate compounds that fail to meet both %RSD and RRF criteria. **Note:** **The CLP SOW OLM03.2 minimum response factor method acceptance criterion differs from the Region I Functional Guidelines initial and continuing calibration minimum response factor validation criterion. If data quality objectives allow for greater variability of data, then an expanded minimum response factor validation criterion should be documented in the EPA-approved site-specific QAPjP or amendment to the QAPjP. If response factors less than 0.05 are allowed, then the validator should ensure that there is sufficient QC data to support the use of low response factors in sample calculations.**	3. **Situation 1:** If any target compound has a %RSD less than or equal to 30.0% and an RRF less than 0.05, then the validator should: a. Estimate (J) positive detects for that affected compound that have acceptable mass spectral identification for all samples associated with the initial calibration. b. Reject (R) non-detects for that affected compound for all samples associated with the initial calibration. **Situation 2:** If any target compound has a %RSD greater than 30.0% **and** an RRF greater than or equal to 0.05, then the validator should: a. Estimate (J) positive detects for that affected compound for all samples associated with the initial calibration. b. Estimate (UJ) non-detects for that affected compound for all samples associated with the initial calibration. c. See D.4, Situation 2 Expanded for additional guidance. **Situation 3:** If any target compound has a %RSD greater than 30.0% **and** an RRF less than 0.05, then the validator should: a. Estimate (J) positive detects for that affected compound that have acceptable mass spectral identification for all samples associated with the initial calibration. b. Reject (R) non-detects for that affected compound for all samples associated with the initial calibration. **Surrogates:** If any surrogate compound fails to meet minimum RRF criteria and/or %RSD criteria, then the validator should use professional judgment to assess the impact of surrogate compound calibration data on the sample results. **See Table VOA/SV-III-1**

C. EVALUATION	D. ACTION
4. Evaluate the cause of a non-linear calibration curve based on 5 or more concentration points.	4. **Situation 2 Expanded:** If the %RSD is greater than 30.0%, and all the initial calibration RRFs for a target compound are greater than or equal to 0.05, then the validator should use professional judgment to determine the need to check the calibration points for the cause of the non-linearity. This is checked by eliminating either the high or the low calibration points and recalculating the %RSD. At the validator's discretion, a more in-depth review to minimize data qualification can be accomplished by considering the following: a. If any target compound has a %RSD greater than 30.0%, and if eliminating either the high point or the low point of the curve does not restore the %RSD to less than or equal to 30.0%, then the validator should: - Estimate (J) positive detects for that affected compound for all samples associated with the initial calibration. - Estimate (UJ) non-detects for that affected compound for all samples associated with the initial calibration. b. If eliminating the high point of the curve restores the %RSD to less than 30.0%, then the validator should: - Accept (A) positive detects in the linear portion of the curve for that affected compound for all samples associated with the initial calibration. - Estimate (J) positive detects at the high end of curve outside of the linear portion for that affected compound for all samples associated with the initial calibration. - Accept (A) non-detects for that affected compound for all samples associated with the initial calibration.

C. EVALUATION	D. ACTION
4. Continued from above.	4. c. If eliminating the low point of the curve restores the %RSD to less than 30.0%, then the validator should: - Accept (A) positive detects in the linear portion of the curve for that affected compound for all samples associated with the initial calibration. - Estimate (J) positive detects at the low end of curve outside linear portion for that affected compound for all samples associated with the initial calibration. - Estimate (UJ) non-detects for that affected compound for all samples associated with the initial calibration. **See Table VOA/SV-III-2**
____ *5. Check and recalculate the RRF and RRF for at least one volatile and semivolatile target compound associated with each internal standard. Verify that the recalculated values agree within 10% of the laboratory reported values.	5. If errors greater than 10% are detected in the RRF calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*6. Check and recalculate the %RSD for at least one volatile and semivolatile target compound associated with each internal standard. Verify that the recalculated values agree within 10% of the laboratory reported values.	6. If errors greater than 10% are detected in the %RSD calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*7. a. Review Standard Preparation Logs (if provided in the data package) to ensure that primary and secondary initial calibration standard concentrations are accurate and traceable to NIST standards.	7. a. If standards preparation data have not been submitted with the data package, then the validator should use professional judgment to determine if standards preparation data are necessary to facilitate the validation of sample data. If necessary, the validator should contact the laboratory to obtain standards preparation information.
* b. Check and recalculate the initial calibration standard concentration for one volatile and one semivolatile target compound (if standards preparation documentation was provided in the data package). Verify that the calculated values agree within 10% of the laboratory reported values.	b. If errors greater than 10% are detected in the standard concentration calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.2, C.5, C.6, C.7.a, C.7.b

Table VOA/SV-III-1:

QUALIFICATION OF VOA/SV ANALYTES BASED ON THE INITIAL CALIBRATION

Sample Results	QC Criterion $\overline{RRF}$ $ 0.05 %RSD # 30.0%	Situation 1 $\overline{RRF}$ <0.05 %RSD # 30.0%	Situation 2** $\overline{RRF}$ $ 0.05 %RSD >30.0%	Situation 3 $\overline{RRF}$ < 0.05 %RSD > 30.0%
Detects	A	J	J	J
Non-detects	A	R	UJ	R

** See Table VOA/SV-III-2 for additional guidance.

Table VOA/SV-III-2:

EXPANDED INITIAL CALIBRATION VOA/SV ANALYTE QUALIFICATIONS

Sample Results	Elimination of High or Low Calibration Points %RSD > 30.0%	Elimination of High Calibration Points %RSD # 30.0% RRF $ 0.05	Elimination of Low Calibration Points %RSD # 30.0% RRF $ 0.05
Detects	J	A: On linear portion of curve J: On high end of curve outside linear portion	A: On linear portion of curve J: On low end of curve outside linear portion
Non-detects	UJ	A	UJ

E. EXAMPLES

Example #1: Situation 1 (Low RRF; Acceptable linearity)

The RRF of an initial calibration for benzene is 0.035 which does not meet the 0.05 acceptance criteria. The %RSD of the calibration points for benzene is 19.0%. Due to the low instrument response for benzene, the validator estimates (J) all the positive benzene detects and rejects (R) the benzene non-detects on the Data Summary Table and notes this problem in the Data Validation Memorandum.

Example #2: (Low RRF; Acceptable linearity; Modified Region I RRF criteria)

The RRF of an initial calibration for acetone is 0.035 and the %RSD is 12.0%. The site-specific EPA-approved QAPjP documents that modified Region I minimum RRF criteria will be used to validate project data. The modified criteria are:

- The mean initial calibration RRF and the continuing calibration RRF for all volatile and semivolatile target and surrogate compounds must be greater than or equal to 0.05 except for the following compounds which must have an initial calibration RRF and a continuing calibration RRF greater than or equal to 0.01: chloromethane, chloroethane, methylene chloride, acetone, carbon disulfide, 1,2-dichloroethane (total), 2-butanone, 1,2-dichloropropane, 4-methyl-2-pentanone, 2-hexanone and surrogates, toluene-d8 and 1,2-dichloroethane-d4.

The validator accepts all acetone positive detects and non-detects in the samples associated with the initial calibration and reports the sample results unqualified on the Data Summary Table. The validator documents the modified data validation criteria in the Data Validation Memorandum.

Example #3: Situation 2 (Acceptable RRF; High RSD - Elimination of high point)

The validator examines the initial calibration data and notes that the %RSD for tetrachloroethene was 60.0% and the RRF was 0.07. Elimination of the high calibration point restored the %RSD to 18.0%. Since linearity was verified for a portion of the tetrachloroethane curve, the validator accepts all positive tetrachloroethene detects on the linear portion of the curve and estimates (J) the positive tetrachloroethene detects on the non-linear portion of the curve. Tetrachloroethene non-detects are accepted. All results are reported on the Data Summary Table and the qualifications are discussed in the Data Validation Memorandum.

Example #4: Situation 2 (Acceptable RRF; High RSD - Elimination of low point)

The validator examines the initial calibration data and notes that

the %RSD for acetone was 70.0% and the RRF was 0.07. Elimination of the low calibration point restored the %RSD to 20.0%. Since linearity was verified for a portion of the acetone curve, the validator accepts all positive acetone detects on the linear portion of the curve and estimates (J) the positive acetone detects on the non-linear portion of the curve. Acetone non-detects are estimated (UJ). All results are reported on the Data Summary Table and the qualifications are discussed in the Data Validation Memorandum.

E. EXAMPLES

Example #5: Situation 3 (Low RRF; High RSD)

The RRF for trichloroethene is 0.029 which is well below the 0.05 acceptance criteria and the %RSD for trichloroethene is 65.0% which is well above the acceptance criteria. Linearity cannot be achieved by eliminating the high or low points. Due to erratic instrument performance, the validator uses professional judgment to estimate (J) positive trichloroethene detects and reject (R) trichloroethene non-detects on the Data Summary Table and discusses sample qualifications in the Data Validation Memorandum.

IV. CONTINUING CALIBRATION

A. OBJECTIVE

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Continuing calibration establishes the daily relative response factors on which target compound quantitation is based and checks the stability of instrument response on a day-to-day basis.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. Continuing calibration standards containing volatile and semivolatile target and surrogate compounds at method-specified concentrations are analyzed at the beginning of each 12-hour analysis period following the analysis of the instrument performance check and prior to the analysis of the field samples, QC samples, and blanks.

2. Continuing calibration standards must be analyzed using the same instrumental conditions which were used to analyze the initial calibration and that will be used to analyze field samples, QC samples, and blanks.

3. The continuing calibration Relative Response Factors (RRFs) for all volatile and semivolatile target and surrogate compounds must be greater than or equal to 0.05.

   The Percent Difference (%D) between the most recent initial calibration RRF and the continuing calibration RRF for all volatile and semivolatile target compounds and surrogate compounds must not exceed ± 25.0 percent.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from continuing calibration anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. a. Verify that the continuing calibration standard was analyzed at the required concentration and frequency, and that the standard was analyzed within 12 hours of the associated GC/MS instrument performance check.	1. a. If the laboratory did not use the required concentration and/or frequency when analyzing the continuing calibration standard or the standard was not analyzed within 12 hours of the associated GC/MS instrument performance check, then the validator should use professional judgment to determine whether the associated sample data should be qualified or rejected.
b. Verify that quantitation was performed using a continuing calibration analyzed within 12 hours of the field samples.	b. If the correct continuing calibration standard was not used to quantitate sample results, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*2. Verify that the same instrument parameters were used for sample and calibration analyses, and that instrument parameters which were utilized met method requirements.	2. If the same method-required instrument parameters (i.e., purge and trap conditions, etc.) were not used for the continuing calibration standards and field sample analyses, then the validator should contact the laboratory. a. If the laboratory is unable to submit a correct continuing calibration, then the validator should determine whether a qualitative analysis is of any benefit by reviewing the project Data Quality Objectives. b. If the data are deemed unusable, then the validator should reject (R) all associated data. The data should be returned to the laboratory and payment denied.

C. EVALUATION	D. ACTION
3. Verify that the continuing calibration was compared to the most recent initial calibration. Verify that RRFs for all volatile and semivolatile target and surrogate compounds are greater than or equal to 0.05 in the continuing calibration. Verify that the %D between initial calibration &&& and continuing calibration RRF for all volatile and semivolatile target and surrogate compounds is less than or equal to ± 25.0%. Evaluate compounds that fail to meet both %D and RRF criteria. **Note:** **The CLP SOW OLM03.2 minimum response factor method acceptance criterion differs from the Region I Functional Guidelines initial and continuing calibration minimum response factor validation criterion. If data quality objectives allow for greater variability of data, then an expanded minimum response factor validation criterion should be documented in the EPA-approved site-specific QAPjP or amendment to the QAPjP. If response factors less than 0.05 are allowed, then the validator should ensure that there is sufficient QC data to support the use of low response factors in sample calculations.**	3. If the continuing calibration was not compared to the most recent initial calibration, then the validator should have the laboratory recalculate %Ds based on the correct initial calibration and resubmit all affected data and forms. **Situation 1:** If any target compound has a %D between the initial calibration and the continuing calibration which is less than or equal to ± 25.0% **and** a continuing calibration RRF less than 0.05, then the validator should: a. Estimate (J) positive detects for that affected compound that have acceptable mass spectral identification for all samples associated with the continuing calibration. b. Reject (R) non-detects for that affected compound for all samples associated with the continuing calibration. **Situation 2:** If any target compound has a %D between the initial and continuing calibration of greater than ± 25.0% **and** a continuing calibration RRF greater than or equal to 0.05, then the validator should: a. Estimate (J) positive detects for that affected compound for all samples associated with the continuing calibration. b. Estimate (UJ) non-detects for that affected compound for all samples associated with the continuing calibration. **Situation 3:** If any target compound has a %D between the initial and continuing calibration of greater than ± 25.0% **and** a continuing calibration RRF less than 0.05, then the validator should: a. Estimate (J) positive detects for that affected compound

C. EVALUATION	D. ACTION
3. Continued from above.	3. Continued from above. **Surrogates:** If any surrogate compound fails to meet minimum RRF criteria and/or %D criteria, then the % surrogate recoveries in the samples, QC samples and blanks associated with the continuing calibration may be biased high or low resulting in unacceptable surrogate recoveries. In this case, the validator should use professional judgment to assess the impact of surrogate compound calibration data on the sample results. **See Table VOA/SV-IV-1**
*4. Check and recalculate the RRF for at least one volatile and semivolatile target compound associated with each internal standard. Verify that the recalculated values agree within 10% of the laboratory reported values.	4. If errors greater than 10% are detected in the RRF calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*5. Check and recalculate the %D for at least one volatile and semivolatile target compound associated with each internal standard. Verify that the recalculated values agree within 10% of the laboratory reported values.	5. If errors greater than 10% are detected in the %D calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*6. a. Review Standard Preparation Logs (if available in the data package) to ensure that primary and secondary continuing calibration concentrations are accurate and traceable to NIST standards.	6. a. If standards preparation data have not been submitted with the data package, then the validator should use professional judgment to determine if standards preparation data are necessary to validate sample data. If necessary, the validator should contact the laboratory to obtain standards preparation information.
* b. Check and recalculate the continuing calibration standard concentration for one volatile and one semivolatile target compound (if standards preparation documentation was provided in the data package). Verify that the calculated values agree within 10% of the laboratory reported values.	b. If errors greater than 10% are detected in the standard concentration calculations, then the validator should perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.2, C.4, C.5, C.6.a, C.6.b

Table VOA/SV-IV-1:

QUALIFICATION OF VOA/SV ANALYTES BASED ON THE CONTINUING CALIBRATION

Sample Results	**QC Criteria** RRF $ 0.05 %D # ± 25.0%	**Situation 1** RRF < 0.05 %D # ± 25.0%	**Situation 2** RRF $ 0.05 %D > ± 25.0%	**Situation 3** RRF < 0.05 %D > ± 25.0%
Detects	A	J	J	J
Non-Detects	A	R	UJ	R

E. EXAMPLES

Example #1: Situation 1 (Low RRF; Acceptable %D)

The RRF for 2-butanone in a continuing calibration is 0.035 and the %D is 10.0%. Due to the low response, the validator estimates (J) all 2-butanone positive detects and rejects (R) all 2-butanone non-detects that are associated with this continuing calibration on the Data Summary Table. The validator discusses the qualification of sample data in the Data Validation Memorandum.

Example #2: (Low RRF; Acceptable %D; Modified Region I RRF criteria)

The RRF for acetone in a continuing calibration is 0.025 and the %D is 12.0%. The site-specific EPA-approved QAPjP documents that modified Region I minimum RRF continuing calibration data validation criteria will be used to validate project data. The modified criteria are:

- The mean initial calibration RRF and the continuing calibration RRF for all volatile and semivolatile target and surrogate compounds must be greater than or equal to 0.05 except for the following compounds which must have an initial calibration RRF and a continuing calibration RRF greater than or equal to 0.01: chloromethane, chloroethane, methylene chloride, acetone, carbon disulfide, 1,2-dichloroethane (total), 2-butanone, 1,2-dichloropropane, 4-methyl-2-pentanone, 2-hexanone and surrogates, Toluene-d8 and 1,2-dichloroethane-d4.

The validator reviews the acetone mass spectra for positive detects in samples and determines that all mass spectral identification criteria are met. The validator accepts all acetone positive detects and non-detects in the samples associated with the continuing calibration and reports the sample results unqualified on the Data Summary Table. The validator documents the modified data validation criteria in the Data Validation Memorandum.

E. EXAMPLES

Example #3: Situation 2 (Acceptable RRF; High %D)

The RRF for methylene chloride in a continuing calibration is greater than 0.05 and the %D between the initial and continuing calibration for methylene chloride is 45.0%. The validator reviews the initial calibration, continuing calibration, and blank data, and determines that an intermittent methylene chloride contamination problem exists in the laboratory which may contribute to the high %D. The validator estimates (J) all methylene chloride positive detects and estimates (UJ) the methylene chloride non-detects in the associated samples on the Data Summary Table. The validator discusses this problem in the Data Validation Memorandum.

Example #4: Situation 3 (Low RRF; High %D)

The RRF for N-nitroso-di-n-propylamine in a continuing calibration is 0.001 and the %D is 110.0%. Due to low and unstable instrument response to N-nitroso-di-n-propylamine, the validator determines that both the quantitation limits and positive detects for N-nitroso-di-n-propylamine are unusable. Therefore, the validator rejects (R) all N-nitroso-di-n-propylamine results that are associated with this continuing calibration on the Data Summary Table. The validator discusses the qualification of sample data in the Data Validation Memorandum.

V. BLANKS

A. OBJECTIVE

The purpose of blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory and/or field activities and to subsequently assess their contribution to measurement error. The criteria for evaluation of laboratory blanks (method blanks and instrument blanks) may be applied to any blank associated with the samples. If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent measurement error associated with the entire data set, or if the problem is an isolated occurrence limited to specific samples.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. The frequency and types of blanks collected and analyzed must support the site-specific Data Quality Objectives (DQOs) as documented in the EPA approved QAPjP or SAP. Different types of blanks may be used to identify the source of potential contamination resulting in analytical and/or sampling measurement error. The following table lists types of blanks, the environment of those blanks, and the possible sources of contamination associated with those blanks:

BLANK	LABORATORY/FIELD	IDENTIFIES CONTAMINATION FROM
Method Blank	Laboratory	Laboratory and Reagents
Instrument Blank	Laboratory	Instrumentation
Storage Blank	Laboratory	Storage Environment
Trip Blank	Field	Transit Environment
Bottle Blank	Field	Sample Container
Equipment Blank (Rinsate)	Field	Sampling Equipment

Note: Aqueous equipment (rinsate) blank results, bottle blank results and trip blank results will be used to determine blank action levels for aqueous samples based on a volume of 1 liter of blank sample. Ideally soil/sediment blanks should be used to determine soil/sediment blank actions for soil/sediment samples based on a known weight of blank sample. However, often aqueous equipment blanks, bottle blanks and trip blanks are collected to evaluate contamination associated with soil/sediment sampling. **Aqueous equipment (rinsate) blank results,**

bottle blank results and trip blank results will not be used to determine blank action levels for non-aqueous samples. Compounds that are present in both the non-aqueous sample and the associated aqueous equipment blank, bottle blank or trip blank will be flagged EB (Equipment Blank), BB (Bottle Blank) or TB (Trip Blank), respectively. The degree of "sampling error" that this flagged sample result represents will be left to the determination of the end user.

2. Method Blanks:

   a. A volatile method blank must be analyzed after the continuing calibration and before any samples, QC samples, or other types of blanks (i.e., storage blanks). The VOA method blank must be analyzed at least once during every 12 hour time period on each GC/MS system used to analyze samples.

   b. A semivolatile method blank must be extracted with each sample delivery group or each 20 samples of similar matrix in each sample delivery group or whenever a sample extract procedure is performed. The method blank must undergo all cleanup procedures performed on samples, i.e., GPC, Silica Gel, etc. used in sample preparation. The semivolatile method blank extract must be analyzed on each GC/MS system used to analyze samples.

3. Instrument Blanks:

   a. An instrument blank must be analyzed after any sample that exceeds the calibration range to check that the blank is free of interference and the system is not contaminated.

   b. For purge and trap volatile organic analysis, an instrument blank must be analyzed in the same purging position as a sample that exceeds the calibration range to check that the blank is free of interference and the purging position is not contaminated.

   c. Instrument blanks and apparatus blanks for each cleanup procedure, including GPC and Silica Gel, etc. used in sample preparation must be analyzed prior to sample analysis.

4. Storage Blanks:

   a. A volatile storage blank vial (in duplicate) must be prepared by the laboratory when the first samples of the sample delivery group are received. The storage blank is stored with the samples and analyzed after all the samples in the sample delivery group have been analyzed.

5. All blanks should be spiked with surrogate compounds and internal standards according to the method. Note: CLP OLM03.2 does not require that the GPC instrument blank be spiked with internal standards or surrogates.

   a. Blank internal standards must meet method internal standard QC acceptance criteria.

   b. Blank surrogate compounds must meet method surrogate compound QC acceptance criteria.

6. No contaminants should be present in the blanks.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from blank anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. **Qualification should be based upon a comparison of the sample concentration(s) with the highest blank concentration associated with the sample delivery group.** However, in cases of specific instrument, storage and/or method blank contamination, the validator should use professional judgment to qualify only those samples associated with that isolated blank contamination. Likewise, the validator may need to apply blank qualifications to a sample delivery group based on associated equipment, trip, or bottle blank data that exists in another sample group data package. **Sample results must not be corrected by subtracting any blank values.**
1. a. Verify that the correct number and type of blanks have been collected and analyzed in accordance with the EPA approved QAPjP or SAP.	1. a. If the correct number and type of blanks have not been collected and analyzed, then the validator should note this deviation from the EPA approved QAPjP or SAP in the Data Validation Memorandum. The validator should use professional judgment to qualify sample data when blank data are absent. When required trip, equipment (rinsate) or bottle blanks are not identified on the chain of custody, then the validator must contact the sampler or site project manager to obtain this information and note this contact on the Blank Analysis validation worksheet.
b. Ascertain if aqueous equipment (rinsate) blanks, aqueous bottle blanks or aqueous trip blanks have been collected with non-aqueous samples to identify sources of field contamination.	b. If positive results are detected in the aqueous

C. EVALUATION	D. ACTION
2. a. Verify that a VOA method blank analysis has been reported per matrix, per concentration level, per extraction batch (for medium-level VOAs only) after each continuing calibration and for each 12-hour time period on each GC/MS system used to analyze samples.	2. a. If VOA method blanks were not analyzed at the required frequency and for each matrix, concentration level, extraction batch (for medium-level VOAs only), and on each GC/MS system used to analyze samples, then the validator should use professional judgment to determine whether the associated sample data should be qualified.
b. Verify that a semivolatile method blank analysis has been reported once per matrix, per concentration level, per extraction technique and SDG, and on each GC/MS system used to analyze sample extracts.	b. If semivolatile method blanks were not analyzed at least once for each matrix, concentration level, extraction technique and batch, and on each GC/MS system used to analyze sample extracts, then the validator should use professional judgment to determine whether the associated data should be qualified.
* c. Verify from the raw data that the extraction and/or analysis dates and times, sample IDs, file IDs, instrument IDs, etc. are accurately reported on the tabulated result forms.	c. If review of the raw data reveals discrepancies and/or transcription errors, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
3. a. Verify from the Blank Summary form and Form Is that a VOA instrument blank was analyzed after each sample that exceeded the instrument calibration range.	3. a. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), then sample analysis results after the high concentration sample must be evaluated for carryover. Professional judgment should be used to determine if instrument cross-contamination has affected any positive compound identification and/or quantitation, and to determine whether the affected sample data should be qualified or rejected. If cross-contamination is suggested, then this should be noted in the Data Validation Memorandum.
* b. Verify from the raw data, the Blank Summary form, and Form Is that a VOA instrument blank was analyzed in the same purging/sparging vessel (i.e., same position in the autosampler) as the sample that exceeded the instrument calibration range.	b. If an instrument blank was not analyzed in the same purging vessel used to analyze a sample that exceeded the instrument calibration range, then sample analysis results generated in that purging vessel after the high concentration sample must be evaluated for carryover. Professional judgment should be used to determine if instrument cross-contamination has affected any positive compound identification and/or quantitation, and to determine whether the affected sample data should be qualified or rejected. If cross-contamination is suggested, then this should be noted in the Data Validation Memorandum.
* c. i. Verify from the raw GPC data that a GPC instrument blank was analyzed after the GPC calibration and prior to sample analysis.	c. i. If a GPC instrument blank was not analyzed at the method-required frequency, then the validator should evaluate the method blank data and use professional judgment to qualify sample
* ii. Verify from the raw Silica Gel data that a Silica Gel Column reagent blank was analyzed prior to sample analysis.	

C. EVALUATION	D. ACTION
4. Verify that a VOA storage blank was analyzed for each sample delivery group and that it was analyzed after all field samples were analyzed.	4. If a VOA storage blank was not analyzed at the correct frequency, then the validator should use professional judgment to accept or qualify sample data.
5. a. Verify that the blank internal standard areas and retention times and surrogate compound recoveries meet method QC acceptance criteria. * b. Check 10% of the raw data for each blank to verify that internal standard areas and retention time data, have been correctly transcribed to tabulated forms and that surrogate compound recovery data have been correctly calculated and transcribed to tabulated forms. Review the blank chromatograms, quantitation reports, and mass spectra to ensure that no false positives or false negatives have been reported.	5. a. If blank internal standard areas and/or retention times and/or surrogate compound recoveries do not meet method QC acceptance criteria, then the validator should use professional judgment in applying blank actions. The possibility of false positives or false negatives being incorrectly reported for the blank should be evaluated. b. If the laboratory has reported a false positive or a false negative and/or has incorrectly transcribed and/or calculated data, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
6. Review the reported results of all associated blanks on the tabulated forms.	6. If a contaminant is found in a blank but <u>not</u> in the sample, no action is taken. If a contaminant is found in both a blank and a sample, then the validator should note this problem in the Data Validation Memorandum and qualify the data according to the following guidance: **Note: If the blank action level for a compound is determined using the value from a bottle blank, equipment blank or trip blank, then the positive values in the bottle, equipment, or trip blank should be reported unqualified on the Data Summary Tables. However, if the blank action is determined using the value from a laboratory blank (e.g., method, storage, or instrument), then the positive values in the trip, bottle, or equipment blanks should be qualified. (See example #6)**
a. Determine if any target compounds are present at or above the quantitation limit/CRQL in any of the blanks.	a. **Target Compound Contaminants at or Above the Quantitation Limit/CRQL:** i. If positive sample results for a compound are greater than 5 times the concentration in any blank (with the exception of the common laboratory contaminants in Section V.C.6.b), then the compound's concentration should be reported as unqualified. ii. If positive sample results for a compound are less than or equal to 5 times the concentration of the compound in any blank (with the exception of the common laboratory contaminants in Section V.C.6.b) but are greater than the quantitation limit, then the sample quantitation limit for

C. EVALUATION	D. ACTION
6. Continued	6. Continued Note: The validator should note that blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" or "10x" criteria, such that a comparison of the total amount of contamination is actually made. (See example #5). Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample data is deemed necessary. If the validator determines that the contamination originates from a source other than the sample, the sample data should be qualified. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurrence can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. In this case, the "5x" rule may not apply; the target compound should be reported as not detected (U), and an explanation of the data qualification rationale should be provided in the Data Validation Memorandum.
b. Determine if any common volatile laboratory contaminants (acetone, methylene chloride, 2-butanone) or any common semivolatile laboratory contaminants (phthalates) are present at or above the quantitation limit/CRQL in any of the blanks.	b. **Common Laboratory Contaminants at or Above the Quantitation Limit/CRQL:** i. If positive sample results for a common laboratory contaminant compound are greater than 10 times the concentration in any blank, then the compound's concentration should be reported as unqualified (See example #3 - 10x rule). ii. If positive sample results for a common laboratory contaminant compound are less than or equal to 10 times the

C. EVALUATION	D. ACTION
6. c. Determine if low level contamination below the quantitation limit exists in any of the blanks.	6. c. **Common Laboratory Contaminants and Target Compounds Below the Quantitation Limit/CRQL:** i. If a positive sample result is reported at less than the quantitation limit and is also less than the blank action level, then the sample quantitation limit should be reported on the Data Summary Tables (See example #2 - 5x rule). ii. If a positive sample result is reported at less than the quantitation limit but is greater than the blank action level, then the estimated sample result should be reported on the Data Summary Tables. iii. If several target compounds are found at low levels, below the quantitation limit, in the laboratory blank(s), it may indicate a systemic problem in the laboratory and should be noted in the Data Validation Memorandum. iv. If low level contamination exists solely in the trip, bottle or equipment (rinsate) blanks, then the validator should notify the sampler. The call should be documented in a telephone log that is included in the Data Validation Memorandum and the date of contact should be noted on the Blank Analysis Worksheet.
d. Determine if gross contamination, greater than 10x CRQL for any analyte, exists in any of the blanks.	d. **Gross Contamination** i. If gross contamination, greater than 10x CRQL for any analyte, exists in any blank, then the validator should reject (R) all affected compounds in samples associated with that blank due to the interference. This serious problem should be discussed in the Data Validation Memorandum.
e. Determine if instrument contamination is isolated to specific sample runs.	

C. EVALUATION	D. ACTION
*6. f. Review the raw data (chromatograms, mass spectra and quantitation reports) to confirm the presence of target and non-target compounds in the blanks and to evaluate the presence of additional contaminants.	6. f. If review of raw data suggests that additional contaminants are present or, conversely, the review indicates false positives have been reported, then the validator should contact the laboratory to obtain additional information and/or have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
7. Evaluate the overall contamination in each type of blank to ascertain probable source(s) of contamination. For example, a contaminated equipment blank might indicate decontamination problems if the method, storage, instrument, and bottle blanks were all clean.	7. If a review of the various types of blanks identifies a potential source of blank contamination, then the validator should discuss this problem in the Data Validation Memorandum. The validator should identify whether the measurement error is a result of either sampling or analytical error or both (see Data Validation Manual p.1).

*** Note: The following subsections are applicable only to a Tier III data validation.**
C.2.c, C.3.b, C.3.c.i, C.3.c.ii, C.5.b, C.6.f

E. EXAMPLES

Example #1: (Bottle blank target compound contaminant $ CRQL, sample result < 5x blank action level)

Carbon disulfide is detected in a water sample at greater than the CRQL, but less than 5x the bottle blank concentration.

5x Rule

	ug/L	
Bottle Blank Result		20
CRQL	10	
Carbon disulfide Sample Result		80
Action Level	100	
Qualified Sample Result		80 U

In this case, the laboratory sample result for carbon disulfide is

less than 100 ug/L (5 x 20) and the validator reports the carbon disulfide result as non-detected at an elevated quantitation limit on the Data Summary Table. Carbon disulfide was not detected in the method blank but was detected at 12 ppb in the trip blank. The validator notes in the Data Validation Memorandum that the bottle blank was contaminated with carbon disulfide, documents the lot number of the sample bottle, and alerts the site project manager regarding a contaminated lot of bottles.

E. EXAMPLES

Example #2: (Instrument blank target compound contaminant < CRQL, sample result < 5x blank action level)

Ethylbenzene is detected in a water sample at less than the CRQL and also less than 5x the instrument blank concentration. The instrument blank contained the highest concentration of ethylbenzene of all blanks analyzed. In addition, all field samples analyzed were associated with the same contaminated instrument blank.

5x Rule

	ug/L	
Instrument Blank Result	5	
CRQL	10	
Ethylbenzene Sample Result	8	J
Action Level	25	
Qualified Sample Result	10	U

In this case, the ethylbenzene sample result is less than 25 ug/L (5 x 5) and is reported non-detected at the CRQL on the Data Summary Table. This problem is noted in the Data Validation Memorandum.

Example #3: (Common laboratory contaminant $ CRQL, sample result > 10x blank action level)

Bis(2-ethylhexyl)phthalate is detected in a water sample at greater than 10x the method blank concentration.

10x Rule

	ug/L	
Blank Result	20	
CRQL	10	
Bis(2-ethylhexyl)phthalate Sample Result	220	
Action Level	200	
Qualified Sample Result	220	

In this case, the bis(2-ethylhexyl)phthalate sample result exceeded the blank action level of 200 ug/L (10 x 20) and the bis(2-ethylhexyl)phthalate sample result is reported unqualified on the Data Summary Table.

Example #4: (Blank target compound contamination in aqueous equipment blank collected with soil samples)

An equipment blank (rinsate) was included in a sample delivery group of soil samples. The validator examines the data and finds that the equipment blank contains 40 ug/L of bis(2-ethyl-hexyl)phthalate. The

validator then reviews all other blank data and finds no further bis(2-ethylhexyl)phthalate contamination. One soil sample contains 60 ug/kg of bis(2-ethylhexyl) phthalate. The validator reports the soil sample result on the Data Summary Table as 60 (EB) to indicate to the end user that sampling error has potentially affected the sample results and notes this information in the Data Validation Memorandum.

E. EXAMPLES

Example #5: (Application of sample weights and volumes with 5x Rule)

Soil sample TAA35 was analyzed as a routine semivolatile soil sample under CLP SOW OLM03.2 and contained 70% solids. The validator reviewed the sample results and found naphthalene (560 ug/kg) and pyrene (460 ug/kg) in sample TAA35. The method blank was found to be contaminated with pyrene (420 ug/kg) and naphthalene (430 ug/kg). These blank results were reported by the laboratory on a dry weight basis and were the maximum levels of contamination found for these compounds in this sample delivery group. The validator determines the blank action level by applying the 5x rule. The method blank action level for pyrene was calculated to be 2100 ug/kg (420 x 5), and the action level for naphthalene was calculated to be 2150 ug/kg (430 x 5).

The validator calculates the sample quantitation limits for naphthalene and pyrene for 30.0 g extracted:

$$\text{naphthalene QL} = \frac{\text{CRQL}}{\%\ \text{solids}} = \frac{330\ \text{ug/kg}}{0.7} = 471\ \text{ug/kg}$$

$$\text{pyrene QL} = \frac{\text{CRQL}}{\%\ \text{solids}} = \frac{330\ \text{ug/kg}}{0.7} = 471\ \text{ug/kg}$$

The validator applies the following action to the naphthalene and pyrene results for sample TAA35:

Naphthalene 5x Rule	ug/kg	Pyrene 5x Rule	ug/kg
Blank Result	430	Blank Result	420
CRQL	471	CRQL	471
Sample Result	560	Sample Result	460 J
Action Level	2150	Action Level	2100
Qualified Sample Result	560 U	Qualified Sample Result	471 U

- The sample quantitation limit for naphthalene is elevated to the sample concentration result on the Data Summary Table and is reported as 560U, since the result falls between the sample quantitation limit and the blank action level.

- The pyrene sample result on the Data Summary Table is replaced with the sample quantitation limit and is reported as 471U, since the positive sample detect of 460 ug/kg is below both the sample quantitation limit and the blank action level.

The validator notes all actions taken in the Data Validation Memorandum.

E. EXAMPLES

Example #6: (Application of laboratory blank action levels to trip blanks)

The method blank for an aqueous batch of volatile samples was contaminated with 25 ug/L of trichloroethene. The trip blank for this batch of samples was contaminated with 22 ug/L of trichloroethene and 15 ug/L of ethylbenzene. Since trichloroethene was detected in both the method blank and the trip blank, the highest detected concentration is used to determine the blank action level. The method blank concentration is, therefore, used to determine the blank action level for trichloroethene.

Trichloroethene	ug/L		Ethylbenzene	ug/L
Method Blank Result	25		Method Blank Result	10 U
Trip Blank Result	22		Trip Blank Result	15
CRQL	10		CRQL	10
Blank Action Level		125(5x25)	Blank Action Level	75 (5x15)

The trichloroethene positive detect in the trip blank is qualified and reported as 22U ug/L on the Data Summary Table. The blank action level for ethylbenzene is determined using the value from the trip blank and, as a result, the ethylbenzene positive detect in the trip blank is reported unqualified as 15 ug/L on the Data Summary Table.

VI. SURROGATE COMPOUNDS

A. OBJECTIVE

Sample matrix effects and laboratory performance on individual samples are assessed by spiking the samples with surrogate compounds prior to extraction and/or analysis and determining their recoveries. Evaluation of surrogate recoveries is not necessarily straightforward. Interfering matrix effects, including high concentrations of target and/or non-target analytes, are frequently outside control of the laboratory and may present relatively unique problems. Therefore, the evaluation and review of the surrogate compound results are frequently subjective, demanding extensive analytical experience and professional judgment. Accordingly, this section consists primarily of guidance with several optional approaches suggested.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Volatile/Semivolatile method QC acceptance criteria listed in Appendices A and B should be used as the default criteria when none exist for the Volatile/Semivolatile analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPjP/SAP or amendment to the QAPjP/SAP.

1. The correct method-required surrogate compounds must be added to all samples, QC samples and blanks at the proper concentrations.

2. a. Recoveries for surrogate compounds in samples, QC samples and blanks must be within the QC acceptance criteria specified in the method.

   b. Recoveries for advisory surrogate compounds in samples, QC samples, and blanks must be greater than or equal to 10%.

3. Volatile samples must be reanalyzed in accordance with method requirements if surrogate compound recoveries are outside the method QC acceptance criteria.

4. Semivolatile samples must be reextracted and/or reanalyzed in accordance with method requirements if surrogate compound recoveries are outside the method QC acceptance criteria.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from surrogate compound anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that the correct compounds were used as surrogate compounds and were added at the required concentrations and frequencies to all samples, QC samples and blanks.	1. a. If surrogate compounds were not added to all samples, QC samples and blanks, were added at the wrong concentration (for example a sample was "double" spiked) or an incorrect compound was used, then the validator should use professional judgment to qualify or reject sample data. b. If surrogate compounds were diluted out of a sample, then the validator should use professional judgment to qualify or reject sample data. Greater than five-fold dilutions result in surrogate recovery data that may be analytically unusable.

C. EVALUATION	D. ACTION
2. a., b., c. Verify that no surrogate compound recovery is outside the method QC acceptance criteria for volatile field and QC samples and verify that no more than one base/neutral surrogate or one acid surrogate is outside method QC acceptance criteria for semivolatile field and QC samples.	2. a. If one surrogate in the VOA fraction or two or more surrogates in the base/neutral or acid fractions have recoveries greater than the upper method QC acceptance limit, then the validator should: i. Estimate (J) all volatile, base/neutral or acid positive detects in the affected sample fraction. ii. Accept all volatile, base/neutral or acid non-detects in the affected sample fraction. b. If one surrogate in the VOA fraction or two or more surrogates in the base/neutral or acid fractions have recoveries greater than or equal to 10% but less than the lower method QC acceptance limit, then the validator should: i. Estimate (J) all volatile, base/neutral or acid positive detects in the affected sample fraction. ii. Estimate (UJ) all volatile, base/neutral or acid non-detects in the affected sample fraction. c. If any surrogate compound in a fraction recovers at less that 10%, then the validator should: i. Estimate (J) all volatile, base/neutral or acid positive detects in the affected sample fraction. ii. Reject (R) all volatile, base/neutral or acid non-detects in the affected sample fraction.

C. EVALUATION	D. ACTION
2. d. Verify that no advisory surrogate compound recovers at less than 10%.	2. d. If any advisory surrogate compound in a fraction recovers at less than 10%, then the validator should use professional judgment to qualify the sample data, taking into account the recoveries of all other surrogate compounds and the compounds of concern at the site.
e. Determine if blank surrogate recovery results meet method QC acceptance criteria.	e. In the special case of a blank analysis with surrogate compound recoveries outside the method QC acceptance criteria, the validator must give special consideration to the validity of the associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if most of the samples including other types of blanks in the batch show acceptable surrogate compound recoveries, then the validator may choose to consider the blank problem to be an isolated occurrence. However, even if this judgment allows some use of the affected data, analytical problems should be noted in the Data Validation Memorandum. All samples that were extracted with or analyzed after an out of control blank should be noted in the Data Validation Memorandum. Also, note in the Data Validation Memorandum if there are potential contractual problems associated with the failure to reextract and/or reanalyze blanks that were outside the method QC acceptance criteria.

C. EVALUATION	D. ACTION
3. For aqueous and low/medium soil volatile samples, verify that if surrogate compound recoveries are outside the method QC acceptance criteria, then the required reanalysis was performed to confirm that the non-compliance was due to sample matrix effects rather than poor laboratory performance.	3. If a laboratory fails to reanalyze a sample which is out of specification, then the sample data should be qualified or rejected according to the guidelines above. The validator should note this method deviation/contractual deficiency in the Data Validation Memorandum.
4. For semivolatile samples, verify that if surrogate compound recoveries are outside the method QC acceptance criteria, then the required reextraction/reanalysis was performed to confirm that the noncompliance was due to sample matrix effects rather than poor laboratory performance.	4. If a laboratory fails to reextract and reanalyze a sample which is out of specification, then the sample data should be qualified or rejected according to the guidelines above. The validator should note this method deviation/contractual deficiency in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*5. a. Check raw data (e.g., chromatograms and quantitation reports) to verify that surrogate recoveries were reported accurately on the Surrogate Recovery Forms.	5. a. If there are any transcription errors, then the validator should contact the laboratory to obtain corrected raw data and forms.
* b. Ten percent of the surrogate compound recovery data should be checked for calculation and/or transcription errors. If errors are detected in this ten percent, then an additional ten percent of the data should be checked. If errors are found in the additional ten percent, then all surrogate compound recovery calculations and transcriptions in the data package should be checked.	b. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.5.a, C.5.b

Table VOA/SV-VI-1:

QUALIFICATION OF VOLATILE/SEMIVOLATILE ANALYTES BASED ON SURROGATE COMPOUND RECOVERIES

	Surrogate Compound Recovery			
Sample Results	**one or more surrogates < 10%**	**one VOA, two B/N or two acid surrogates 10% # %Rec < LL**	**all VOA, one B/N or one acid surrogate LL # %Rec # UL**	**one VOA, two B/N or two acid surrogates > UL**
Detects	J	J	A	J
Non-detects	R	UJ	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

E. EXAMPLES

Example #1: (Two low acid surrogate recoveries - one of which recovered at less than 10%)

Semivolatile aqueous sample SA125, analyzed by CLP SOW OLM03.2, recovered two acid surrogate compounds, phenol-d_5 and 2-fluorophenol, below the method QC acceptance criteria. In addition, the phenol-d_5 recovered at less than 10%. All other surrogate recoveries met QC criteria. The following table lists the surrogate spike recoveries and the method QC acceptance criteria:

Sample No.	Phenol-d_5 % Recovery	QC Acceptance Criteria (aqueous)	2-Fluorophenol % Recovery	QC Acceptance Criteria (aqueous)
SA125	8	10-110	15	21-110

The sample was reextracted and reanalyzed with similar results. The validator examines the PE sample results, and determines that the laboratory accurately prepared and analyzed the QC samples. Also, all internal standard areas were acceptable and the MS/MSD results for sample SA126 did not show a low bias for acid compounds. Therefore, the validator estimates (J) positive detects and rejects (R) non-detects for the acid fraction of sample SA125 on the Data Summary Table. The validator notes in the Data Validation Memorandum that the low recoveries may be due to matrix interferences specific to sample SA125.

E. EXAMPLES

Example #2: (One low volatile surrogate recovery)

Volatile drinking water sample SA925, analyzed by the Region I 524.2 method-Revision 8.0, had one surrogate compound, 1,2-dichlorobenzene-d_4, recover below the method QC acceptance criteria. The other surrogate compound (1,2-dichloroethane-d_4) recovered within the method QC acceptance criteria. The following table lists the surrogate spike recovery and the QC acceptance criteria:

Sample No.	1,2-Dichlorobenzene-d_4 % Recovery	QC Acceptance Criteria (drinking water)
SA925	45	80-120

The sample was reanalyzed 22 days past the holding time. 1,2-Dichlorobenzene-d_4 recovered at 52% in the reanalysis. The validator reports SA925 sample results from the initial analysis because the reanalysis results may be biased low due to the exceeded holding time. The validator reviews the MS/MSD results for sample SA928 and determines that there is no indication of matrix bias in this data set. The validator estimates (J) positive detects and estimates (UJ) non-detects in sample SA925 on the Data Summary Table and notes in the Data Validation Memorandum that the low recovery may be due to matrix interferences specific to SA925.

Example #3: (One slightly low acid and one slightly low base/neutral surrogate recovery)

Semivolatile soil sample SA225, analyzed by CLP SOW OLM03.2, had one acid surrogate compound, 2,4,6-tribromophenol, and one base/neutral surrogate compound, 2-fluorobiphenyl, recover below the method QC acceptance criteria but above 10%. The following table lists the surrogate spike recoveries and the method QC acceptance criteria:

Sample No.	2,4,6-Tribromophenol % Recovery	QC Acceptance Criteria (soil/sediment)	2-Fluorobiphenyl % Recovery	QC Acceptance Criteria (soil/sediment)
SA225	16	19-122	22	30-115

Reanalysis was not contractually required because only one acid surrogate and only one base/neutral surrogate exceeded method QC acceptance criteria. The validator reviews the MS/MSD results for sample SA228 and determines that there is no indication of matrix bias in this data set. The validator examines all surrogate recoveries, including the advisory surrogates in the sample, and determines that validation criteria were met. The validator reports the sample results

unqualified on the Data Summary Table.

E. EXAMPLES

Example #4: (Two slightly low acid surrogate recoveries)

Semivolatile soil sample SA882, analyzed by CLP SOW OLM03.2, had two acid surrogate compounds, phenol-d_5 and 2-fluorophenol, recover below the method QC acceptance criteria. All other surrogate recoveries met method QC acceptance criteria. The following table lists the surrogate spike recoveries and the method QC acceptance criteria:

Sample No.	Phenol-d_5 % Recovery	QC Acceptance Criteria (soil/sediment)	2-Fluorophenol % Recovery	QC Acceptance Criteria (soil/sediment)
SA882	20	24-113	18	25-121

The sample was reextracted and reanalyzed with similar results. The validator reviews the MS/MSD results for sample SA880 and determines that there is no indication of matrix bias in this data set. The validator estimates (J) positive detects and estimates (UJ) non-detects for the acid fraction of sample SA882 on the Data Summary Table and notes in the Data Validation Memorandum that the low recovery may be due to matrix interferences specific to sample SA882.

Example #5: (One advisory base/neutral surrogate with 0% recovery)

1,2-dichlorobenzene is a contaminant of concern at Site XX. Semivolatile water sample SA335, analyzed by CLP SOW OLM03.2, had advisory surrogate compound, 1,2-dichlorobenzene-d_4, recover at 0%. All of the remaining surrogate compounds and advisory surrogate compounds had recoveries which were within method QC acceptance criteria. The validator reviews the MS/MSD results for sample SA336 and determines that there is no indication of matrix bias in this data set. The validator uses professional judgment to reject (R) the analyte of concern, 1,2-dichlorobenzene, and to reject (R) the other dichlorobenzene isomers in sample SA335, based upon their chemical similarity to the advisory surrogate. The validator reports the qualified results on the Data Summary Table and notes in the Data Validation Memorandum that the low recovery may be due to matrix interferences specific to sample SA335 or poor laboratory technique during the sample extraction and/or cleanup procedures.

E. EXAMPLES

Example #6: (One high volatile surrogate recovery)

Volatile soil sample SA966, analyzed using SW-846 Method 8260, recovered one surrogate above the method QC acceptance criteria. The following table lists the surrogate percent recoveries and method QC acceptance criteria:

Sample Number	Toluene-d_8 % Recovery	QC Acceptance Criteria
SA966	128	81 - 117

The sample was reanalyzed within holding time with similar results. The validator reviews the MS/MSD results for sample SA960 and determines that there is no indication of matrix bias in this data set. The validator estimates (J) positive detects and accepts (A) non-detects in the associated sample. The validator reports qualified data on the Data Summary Table and notes sample qualifications in the Data Validation Memorandum.